

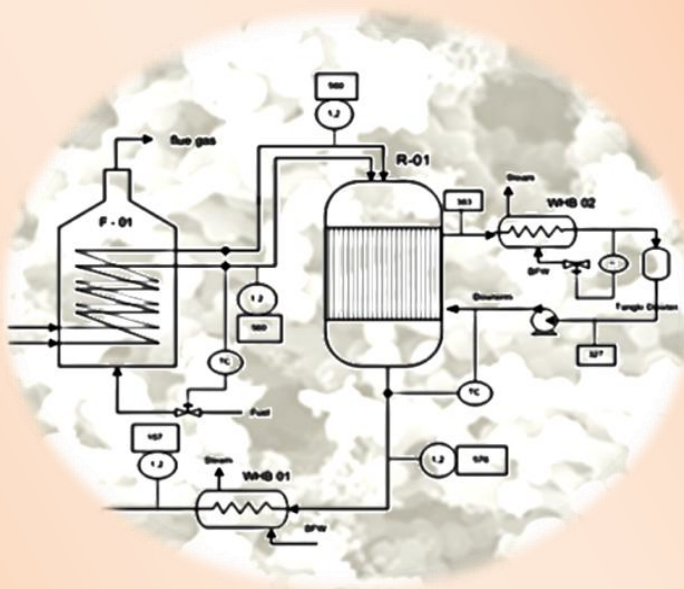


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### INTERNATIONAL CONFERENCE ON CHEMICAL AND MATERIAL ENGINEERING



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# Table of Papers

## Invited Speakers

- [IS – 01](#) **PROF. DR. MATHIAS ULBRICHT**  
UDuE, Germany
- [IS – 02](#) **PROF. DR. PURWANTO**  
Diponegoro University, Indonesia
- [IS – 03](#) **IR. R. GUNUNG SARDJONO HADI, MT**  
PT Pertamina Gas, Indonesia
- [IS – 04](#) **DRS. HARDIONO, MComm**  
BP Migas, Indonesia
- [IS – 05](#) **ICHSAN, MSC, PDENG**  
PT Maris Sustainable Indonesia
- [IS – 06](#) **PROF. DR. HADI NUR**  
UTM, Malaysia

## Oral Presentation

### BRE – BIOPROCESS AND RENEWABLE ENERGY

- [BRE – 01](#) **ENZYMATIC HYDROLYSIS OF ALKALINE PRETREATED COCONUT COIR**  
Akbarningrum Fatmawati<sup>a</sup>, Rudy Agustriyanto<sup>a</sup>, Carolina Adhelia<sup>a</sup>, Jovita Paulina<sup>a</sup>, Yusnita Liasari<sup>b</sup>  
<sup>a</sup> Chemical Engineering Department, Faculty of Engineering, Surabaya University, INDONESIA  
<sup>b</sup> Faculty of Biotechnology, Surabaya University, INDONESIA
- [BRE – 02](#) **OPTIMIZATION OF STREPTOMYCES SP.A11 MEDIUM CULTIVATION ON CYCLO(TYROSYL-PROLYL) PRODUCTION USING THE RESPONSE SURFACE METHODOLOGY**  
Rofiq Sunaryanto  
Center of Biotechnology, Badan Pengkajian dan Penerapan Teknologi (BPPT), INDONESIA
- [BRE – 03](#) **ENHANCEMENT OF BIOMASS PRODUCTION FROM SPIRULINA SP CULTIVATED IN POME MEDIUM**  
Hadiyanto, Muhamad Maulana Azimatun Nur, Ganang Dwi H  
Center of Biomass and Renewable Energy, Department of Chemical Engineering Diponegoro University, INDONESIA
- [BRE – 04](#) **ETHANOL PRODUCTION FROM NON FOOD TUBERS OF ILES-ILES (AMORPHOPHALLUS CAMPANULATUS) USING HYDROLYZES BY COMMERCIAL ENZYMES (A AND B AMYLASE) AND FERMENTATION BY SACCHAROMICES CEREVISEAE**  
Kusmiyati<sup>a,b</sup>, Asha Tridayana<sup>a</sup>, Nurul Widya FP<sup>a</sup>, Tri Utami<sup>a</sup>  
<sup>a</sup> Chemical Engineering Department, Faculty of Engineering, Muhammadiyah Surakarta University INDONESIA  
<sup>b</sup> Renewable Energy Research Centre, Muhammadiyah Surakarta University - Surakarta INDONESIA
- [BRE – 05](#) **SYNTHESIS OF ZEOLITE PELLETS FROM NATURAL ZEOLITE AND STARCH AS ADSORBENT FOR FUEL GRADE BIOETHANOL PRODUCTION**  
Anwar Ma'ruf and Neni Damajanti  
Chemical Engineering Department, Faculty of Engineering, Muhammadiyah University of Purwokerto, INDONESIA

- BRE – 06** **QUALITY IMPROVEMENT (FIBER CONTENT AND PROTEIN DIGESTIBILITY VALUE) OF CASSAVA PEEL BY FERMENTATION USING TAPE YEAST WITH VITAMIN B SUPPLEMENTATION**  
 Wikanastri Hersoelistyorini<sup>a</sup>, Cahya S. Utama<sup>b</sup>  
<sup>a</sup> Food Technology Study Program, University of Muhammadiyah Semarang, INDONESIA  
<sup>b</sup> Department of Animal Feed and Nutrition, Diponegoro University, INDONESIA
- BRE – 07** **THE DETERMINATION OF SALINITY PROFILE AND NUTRITION (NAH<sub>2</sub>PO<sub>4</sub>) PROFILE IN UTILIZING NANNOCHLOROPSIS OCULATA TO GAIN MAXIMUM LIPID**  
 Elida Purba, Kenjiro Parsaulian Siburian  
 Chemical Engineering Department, Faculty of Engineering, University of Lampung, INDONESIA
- BRE – 08** **DESIGN PROCESS OF ETHANOL PRODUCTION BY EXTRACTIVE -FERMENTATION TO INCREASE THE YIELD AND PRODUCTIVITY OF ETHANOL**  
 Ayu Ratna Permanasari, Ririn Indriani AR, Tri Widjaja, Ali Altway  
 Chemical Engineering Department, Faculty of Industrial Technology, Sepuluh Nopember Institut of Technology, INDONESIA
- BRE – 09** **TRANSESTERIFICATION OF VEGETABLES OIL USING SUB-AND SUPERCRITICAL METHANOL**  
 Nyoman Puspa Asri<sup>a,d</sup>, Siti Machmudah<sup>a,b</sup>, Wahyudiono<sup>c</sup>, Suprpto<sup>a</sup>, Kusno Budikarjono<sup>a</sup>, Achmad Roesyadi<sup>a</sup>, Mitsuru Sasaki<sup>c</sup>, Motonobu Goto<sup>b</sup>  
<sup>a</sup> Chemical Engineering Department, Industrial Technology Faculty, Sepuluh Nopember Institute of Technology, Surabaya, INDONESIA 60111  
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<sup>c</sup> Graduate School of Science and Technology, Kumamoto University, JAPAN  
<sup>d</sup> Chemical Engineering Department, Faculty of engineering, WR Supratman University, Surabaya, INDONESIA
- BRE – 10** **THE INFLUENCE OF CATALYSTS TO SELECTIVITY OF PRODUCT OF PALM OIL CRACKING**  
 Achmad Roesyadi, Danawati Hariprajitno, Nurjannah, Santi Dyah Savitri  
 Chemical Reaction Engineering Laboratory, Department Of Chemical Engineering Department Of Chemical Engineering, INDONESIA
- BRE – 11** **BIO-LUBRICANTS DEVELOPMENT: POTENTIAL USE OF BORON-CONTAINING ADDITIVES**  
 Dicky Dermawan, Dyah Setyo Pertiwi  
 Chemical Engineering Department, Faculty of Industrial Technology, Itenas, INDONESIA
- BRE – 12** **UTILIZATION POTENCY OF EXTRACELLULAR POLYMERIC SUBSTANCE AS INDUSTRIALS BIOSORBENT AND ION EXCHANGE RESIN**  
 Zainus Salimin<sup>a</sup>, Pungky Ayu Artiani<sup>a</sup>, Junaidi<sup>b</sup>, and Wawan<sup>b</sup>  
<sup>a</sup> Radioactive Waste Technology Center, National Nuclear Energy Agency – BATAN, INDONESIA  
<sup>b</sup> Study Program of Environmental Engineering, Faculty of Engineering, Diponegoro University, INDONESIA
- BRE – 13** **IMMOBILIZATION OF COW RUMEN FLUID CELLULASE BY ENTRAPMENT IN CALCIUM ALGinate BEADS**  
 Indah Hartati<sup>a</sup>, Laeli Kurniasari<sup>a</sup>, and Agnes Budiarti<sup>b</sup>  
<sup>a</sup> Department of Chemical Engineering, Faculty of Engineering, Wahid, INDONESIA  
<sup>b</sup> Department of Pharmacy, Faculty of Pharmacy, Wahid Hasyim University, INDONESIA
- BRE – 14** **OPTIMIZATION PROCESS OF BIODIESEL PRODUCTION FROM NYAMPLUNG SEED (CALOPHYLLUM INOPHYLLUM L) USING IN SITU PROCESS AND ULTRASONIC ASSISTED**  
 Widayat, Abdullah, Kanevi Octova Paradita and Elsanta Monaliza Tungga D  
 Department of Chemical Engineering Faculty of Engineering Diponegoro University, INDONESIA
- BRE – 15** **EFFECT OF PHENYLACETIC ACID ADDITION ON PRODUCTIVITY OF PENICILLIUM CHRYSOGENUM IN PENICILLIN G PRODUCTION USING PILOT SCALE REACTOR**  
 Amila Pramisandi, Rofiq Sunaryanto, Suyanto  
 Center for the Application of Biotechnology, BPPT, INDONESIA
- BRE – 16** **STUDY OF ENZYMATIC HYDROLYSIS OF DILUTE ACID PRETREATED COCONUT HUSK**  
 Rudy Agustriyanto, Akbarningrum Fatmawati, Maria Angelina, Raissa Monica  
 Surabaya University, INDONESIA



- BRE – 17 KINETICS OF ETHANOL PRODUCTION FROM WHEY BY FEMENTATION USING KLUYVEROMYCES MARXIANUS**  
 Dessy Ariyanti<sup>ab</sup>, Desiyantri Siti Pinundi<sup>a</sup>, Apsari Puspita Aini<sup>a</sup>, Hadiyanto<sup>ab</sup>, Djoko Murwono<sup>a</sup>  
<sup>a</sup> Chemical Engineering Department, Faculty of Engineering, Diponegoro University, INDONESIA  
<sup>b</sup> Center of Biomass and Renewable Energy (C-BIORE), Faculty of Engineering, Diponegoro University, INDONESIA
- BRE – 18 POTENTIAL OF  $\text{SO}_4^{2-}$  / ZnO ACID CATALYST FOR HETEROGENEOUS TRANSESTERIFICATION OF VEGETABLE OIL TO BIODIESEL**  
 I. Istadi, Didi D Anggoro, Luqman Buchori, Inshani Utami, and Roikhatus Solikhah  
 Laboratory of Energy and Process Engineering, , Chemical Reaction Engineering an Catalysis Group, Department of Chemical Engineering, Diponegoro University, INDONESIA
- BRE – 19 A SIMPLE METHOD FOR EFFICIENT EXTRACTION AND SEPARATION OF C-PHYCOCYANIN FROM SPIRULINA PLATENSIS**  
 Noer Abyor Handayani<sup>ab</sup>, Hadiyanto<sup>ab</sup>, Melinda<sup>a</sup>, Inggar<sup>a</sup>, Amin Nugroho<sup>a</sup>  
<sup>a</sup> Chemical Engineering Department, Faculty of Engineering, Diponegoro University, INDONESIA  
<sup>b</sup> Center of Biomass and Renewable Energy (C-BIORE), Faculty of Engineering, Diponegoro University, INDONESIA
- BRE – 20 STUDY ON SLAUGHTERHOUSE WASTES POTENCY AND CHARACTERISTIC FOR BIOGAS PRODUCTION**  
 Budiyo<sup>a</sup>, I Nyoman Widiasta<sup>a</sup>, Seno Johari<sup>b</sup>, Sunarso<sup>c</sup>  
<sup>a</sup> Department of Chemical Engineering, Diponegoro University, INDONESIA  
<sup>b</sup> Faculty of Animal Science and Agriculture, Diponegoro University, INDONESIA
- BRE – 21 STUDY ON PRODUCTION PROCESS OF BIODIESEL FROM RUBBER SEED (HEVEA BRASILIENSIS) BY IN SITU TRANSESTERIFICATION METHOD WITH ALKALINE CATALYZED**  
 Widayat<sup>a,b</sup>, Agam Duma Kalista Wibowo<sup>a</sup>, Hadiyanto<sup>a,b</sup>  
<sup>a</sup> Magister of Chemical Engineering, Diponegoro University, INDONESIA  
<sup>b</sup> Central of Biomass and Renewable Energy, INDONESIA
- BRE – 22 THERMOGRAVIMETRY CHARACTERISTICS OF MSW CHAR BRIQUETTE COMBUSTION**  
 Dwi Aries Himawanto<sup>a</sup>, Indarto<sup>b</sup>, Harwin Saptoadi<sup>b</sup>, Tri Agung Rohmat<sup>b</sup>  
<sup>a</sup> Mechanical Engineering Department Faculty of Engineering, Sebelas Maret University, INDONESIA  
<sup>b</sup> Mechanical and Industrial Engineering Department Faculty of Engineering, Gadjah Mada University, INDONESIA
- BRE – 23 PERFORMANCE OF SULFONATED POLY ETHER-ETHER KETONE (SPEEK) AND NAFION MEMBRANE IN PALM OIL MILL EFFLUENT MICROBIAL FUEL CELL**  
 Nur Dianaty Nordina Abdul Halim Mohd Yusof<sup>a,b,c</sup>, Ahmad Fauzi Ismail<sup>a,d</sup>, Mohd Noorul Anam Mohd Norddin<sup>a,d</sup>  
<sup>a</sup> Faculty of Chemical Engineering, Universiti Teknologi Malaysia, MALAYSIA  
<sup>b</sup> Advanced Membrane Technology Research Centre, Universiti Teknologi Malaysia, MALAYSIA  
<sup>c</sup> Department of Biology, Massachusetts Institute of Technology, UNITED STATES OF AMERICA  
<sup>d</sup> Faculty of Petroleum and Renewable Engineering, Universiti Teknologi Malaysia, MALAYSIA.
- BRE – 24 SURFACE MODIFICATION OF POLYETHERSULFONE WITH POLYVINYLPIRROLIDONE-IODINE VIA PHASE INVERSION AND UV PHOTOGRATING FOR ANTIBACTERIAL APPLICATIONS**  
 Abdul Halim Mohd Yusof<sup>a,b</sup>, Devanai Kannan<sup>a</sup>  
<sup>a</sup> Department of Bioprocess Engineering, Faculty of Chemical Engineering;Universiti Teknologi Malaysia, MALAYSIA  
<sup>b</sup> Department of Biology, Massachusetts Institute of Technology, UNITED STATES of AMERICA
- BRE – 25 ESTERIFICATION OF JATROPHA OIL TO BIODIESEL OVER  $\text{SiO}_2$ -PHOSPHOTUNGSTIC ACID HETEROGENEOUS CATALYST**  
 Nur Hidayati, Aning Tri Aisyah, Ike Sambung Sari and Prinda Widayarni  
 Chemical Engineering Department, Universitas Muhammadiyah Surakarta, INDONESIA

- [MSD – 01](#) **CHARACTERIZATION OF SAGO STARCH AND STUDY OF LIQUEFICATION PROCESS ON HIGH FRUCTOSE SYRUP PRODUCTION**  
Anastasia Prima Kristijarti, Tony Handoko, Cindy Adelia, Lucy Andrea  
*Chemical Engineering Department, Faculty of Industrial Engineering, Parahyangan Catholic University, INDONESIA*
- [MSD – 02](#) **POLYMER SOLAR CELLS: EFFECTS OF ANNEALING TREATMENT AND POLYMER BLENDS ON I-V CHARACTERISTICS**  
Erlyta Septa Rosa and Shobih  
*Research Center for Electronics and Telecommunication, Indonesian Institute of Sciences (PPET-LIPI), INDONESIA*
- [MSD – 03](#) **THE OPTIMIZATION OF DIPHENYL METHANE DIISOCYANATE POLYMERIZATION PROCESS WITH THE USED FRYING OIL POLYALCOHOL TO FOAM POLYURETHANE USING RSM**  
Faleh Setia Budi<sup>a</sup> and Didi Dwi Anggoro<sup>b</sup>  
<sup>a</sup> *Food Science and Technology Department, Faculty of Agricultural Engineering and Technology, IPB, INDONESIA*  
<sup>b</sup> *Chemical Engineering Department, Engineering Faculty UNDIP, INDONESIA*
- [MSD – 04](#) **NUMERICAL ANALYSIS OF A JOURNAL BEARING WITH CHEMICAL ROUGHNESS**  
Mohammad Tauviqirrahman<sup>a,b</sup>, Muchammad<sup>a</sup>, Jamari<sup>b</sup>, and Dik J. Schipper<sup>a</sup>  
<sup>a</sup> *Laboratory for Surface Technology and Tribology, Faculty of Engineering Technology, University of Twente, THE NETHERLANDS*  
<sup>b</sup> *Department of Mechanical Engineering, University of Diponegoro, INDONESIA*
- [MSD – 05](#) **INVESTIGATION ON CENTRIFUGAL PUMP SHAFT: A COMPARISON STUDY OF THE SME AND THE IMPORTED PRODUCT**  
Rifky Ismail, Sugiyanto, Mohammad Tauviqirrahman and Jamari  
*Mechanical Engineering Department, Faculty of Engineering, Diponegoro University, Jl. Prof Sudharto, SH-Tembalang, Semarang INDONESIA*
- [MSD – 06](#) **PREPARATION OF NANOPARTICLE SILICA FROM SILICA SAND AND QUARTZITE BY ULTRAFINE GRINDING**  
Agus Wahyudi<sup>a</sup>, Teguh Nurasi<sup>b</sup> And Siti Rochani<sup>a</sup>  
<sup>a</sup> *R&D Centre for Mineral and Coal Technology, INDONESIA*  
<sup>b</sup> *Department of Chemistry, University of Jenderal Achmad Yani, INDONESIA*
- [MSD – 07](#) **APPLICATION OF TiO<sub>2</sub> FOR SELF CLEANING IN WATER BASED PAINT WITH POLYETHYLENE GLYCOL (PEG) AS DISPERSANT**  
Nining Kusmahetiningsih, Dyah Sawitri  
*Departement of Engineering Physics, Faculty of Industrial Engineering, Sepuluh Nopember Institute of Technology, INDONESIA*
- [MSD – 08](#) **USING SELF CLEANING TiO<sub>2</sub> PHOTOCATALYST IN THE MAKING OF CERAMIC TILES TO DECREASE AMMONIUM CONCENTRATION AND BACTERIUM GROWTH**  
Ana Hidayati M<sup>a</sup>, Sri Darmawati<sup>a</sup>, Muh. Amin<sup>b</sup>  
<sup>a</sup> *Health Analyst Department, Nursing and Health Faculty, University of Muhammadiyah Semarang, INDONESIA*  
<sup>b</sup> *Mechanical Engineering Department, Engineering Faculty, University of Muhammadiyah Semarang, INDONESIA*
- [MSD – 09](#) **THE EFFECT OF POLYANILINE ADDITION ON THE PROPERTIES OF CARBON-BASED POLYPROPYLENE COMPOSITE**  
Lies A. Wisojodharmo, Dewi Kusuma Arti, Eniya Listiani Dewi  
*Center for Materials Technology, Agency for the Assessment and Application Technology (BPPT), INDONESIA*

- MSD – 10** **COMPUTATIONAL ANALYSIS OF WALL SLIP AND CAVITATION IN LUBRICATED SLIDING SYSTEMS**  
 Muchammad<sup>a</sup>, M. Tauviquirrahman<sup>a</sup>, J. Jamari<sup>b</sup>, and D.J. Schipper<sup>a</sup>  
<sup>a</sup> *Laboratory for Surface Technology and Tribology, Faculty of Engineering Technology, University of Twente, THE NETHERLANDS*  
<sup>b</sup> *Department of Mechanical Engineering, University of Diponegoro, INDONESIA*
- MSD – 11** **FRICTION ANALYSIS ON SCRATCH DEFORMATION MODES OF VISCO-ELASTIC-PLASTIC MATERIALS**  
 Budi Setiyana<sup>a</sup>, I. Syafaat<sup>a</sup>, J. Jamari<sup>b</sup>, and D.J. Schipper<sup>a</sup>  
<sup>a</sup> *Laboratory for Surface Technology and Tribology, Faculty of Engineering Technology, University of Twente, THE NETHERLANDS*  
<sup>b</sup> *Department of Mechanical Engineering, University of Diponegoro, INDONESIA*
- MSD – 12** **PREDICTION OF SLIDING WEAR OF ARTIFICIAL ROUGH SURFACES**  
 Imam Syafa'at<sup>a</sup>, Budi Setiyana<sup>a</sup>, Rifky Ismail<sup>a</sup>, Eko Saputra<sup>a</sup>, J. Jamari<sup>b</sup>, D.J. Schipper<sup>a</sup>  
<sup>a</sup> *Laboratory for Surface Technology and Tribology, Faculty of Engineering Technology, University of Twente, THE NETHERLANDS*  
<sup>b</sup> *Department of Mechanical Engineering, University of Diponegoro, INDONESIA*
- MSD – 13** **THE APPLICATION OF B-CYCLODEXTRIN AND POLYETHYLENE GLYCOL 6000 IN THE MICRONISATION OF DRUG – POLYMER COMPOSITE WITH PARTICLE FROM GAS SATURATED SOLUTIONS (PGSS) METHOD**  
 Putu Riani Pradnyandari, Rizky Tetrisyanda, Prida Novarita Trisanti, Firman Kurniawansyah, and Sumarno  
*Chemical Engineering Department, Faculty of Industrial Technology, Institute of Technology Sepuluh Nopember (ITS), INDONESIA*
- MSD – 14** **THE EFFECT OF SONICATION ON THE CHARACTERISTIC OF CHITOSAN**  
 Azra Yuliana, Linggar S. Pradeckta, Emma Savitri, Anita R. Handaratri, Sumarno  
*Chemical Engineering Department, Faculty of Industry Technology, Sepuluh Nopember Institut of Technology Surabaya, INDONESIA*
- MSD – 15** **EFFECTS OF CHAIN EXTENDER TO THE STRUCTURE OF CASTOR OIL-BASED POLYURETHANE FOAM**  
 Edhi Pratondo, Adityo Wahyu Hanggoro, Eva Oktavia Ningrum, Sumarno  
*Chemical Engineering Department, Faculty of Industrial Technology, Sepuluh Nopember Institute of Technology, INDONESIA*
- MSD – 16** **THE SYNTHESIS OF CARBOXYMETHYL AMYLOSE GRAFTED POLYACRYLAMIDE AND ITS APPLICATION IN DRUG RELEASE ASPIRIN**  
 Noor Hidayati, Naila Amanda, Karsono Samuel Padmawijaya, Firman Kurniawansyah, and Sumarno  
*Chemical Engineering Department, Faculty of Industry Technology, Sepuluh Nopember Institut of Technology Surabaya, INDONESIA*
- MSD – 17** **EFFECT OF TWEEN 80'S EMULSIFIER CONCENTRATION WITH SPONTANEOUS DIFFUSION METHOD ON STABILITY SOLUTION TEGERAN'S NANOEMULSION NATURAL DYES**  
 Heny Herawati<sup>a</sup>, Sri Yuliani<sup>a</sup>, Meika Syahbana Rusli<sup>b</sup>, Ratih Purnamasari<sup>b</sup>  
<sup>a</sup> *Indonesia Center For Agricultural Postharvest Research and Development, INDONESIA*  
<sup>b</sup> *Bogor Agricultural University, Agricultural Technology Faculty, IPB, INDONESIA*
- MSD – 18** **NANO CRYSTALLINE STARCH AND ITS ALTERNATIF IMPLEMENTATION**  
 Heny Herawati  
*Indonesia Center For Agricultural Postharvest Research and Development, INDONESIA*
- MSD – 19** **COMPOSITE SPEEK WITH NANOPARTICLES FOR FUEL CELL'S APPLICATIONS: REVIEW**  
 Arief Rahman Hakim  
*Department of Chemical Engineering, Diponegoro University, INDONESIA*
- MSD – 20** **COMPARISON ON MODELLING OF DRYING KINETICS OF GRANULAR POLYMERS PA6 BY DIFFUSION MODELS AND NORMALIZATION METHOD**  
 Suherman<sup>a</sup>, Mirko Peglow<sup>b</sup>, and Evangelos Tsotsas<sup>b</sup>  
<sup>a</sup> *Chemical Engineering Department, Faculty of Engineering, Diponegoro University, Jl. Prof Sudharto, SH-Tembalang, Semarang, INDONESIA*  
<sup>b</sup> *Thermal Process Engineering, Otto-von-Guericke-University, Universitätsplatz 2, 39106 Magdeburg, GERMANY*

- MSD – 21 **CURRENT DENSITY PERFORMANCES IN POLY ETHER ETHER KETON MEMBRANE FOR DIRECT METHANOL FUEL CELLS**  
Tutuk Djoko Kusworo<sup>a</sup>, E. L. Dewi<sup>b</sup>, D. K. Arti<sup>a</sup>, A. Dhuhita<sup>a</sup>, A. Fauzi Ismail<sup>c</sup> and M.N.A. Mohd Norddin<sup>c</sup>  
<sup>a</sup> Chemical Engineering Department, Engineering Faculty, Diponegoro University, INDONESIA  
<sup>b</sup> Agency of Assessment and Application of Technology  
<sup>c</sup> Advanced Membrane Technology Research Centre, Faculty of Chemical and Natural Resources Engineering, Universiti Teknologi Malaysia, MALAYSIA
- MSD – 22 **PREPARATION AND CHARACTERIZATION OF ZEOLITE MEMBRANE**  
Aprilina Purbasari<sup>a</sup>, Titik Istirokhatun<sup>b</sup>, Heny Kusumayanti<sup>a</sup>, Ariestya M. Devi<sup>a</sup>, Lulluil Mahsunah<sup>a</sup>, Heru Susanto<sup>a</sup>  
<sup>a</sup> Chemical Engineering Department, Faculty of Engineering, Diponegoro University, INDONESIA  
<sup>b</sup> Environmental Engineering Department, Faculty of Engineering, Diponegoro University, INDONESIA
- MSD – 23 **EFFECT OF SOIL BURIAL ON THE MECHANICAL PROPERTIES OF HEAT TREATED AND UNTREATED RED BALAU SAW DUST FILLED LDPE COMPOSITES.**  
Ruth Anayimi Lafia-Araga, Aziz Hassan, Rosiyah Yahya, Normasmira Abd. Rahma  
Chemistry Department, Faculty of Science, University of Malaya, Malaysia
- MSD – 24 **IMPACT AND DSC PROPERTIES OF HEAT TREATED AND UNTREATED RED BALAU (SHOREA DIPTEROCARPACEAE)/LDPE COMPOSITES**  
Aziz Hassan, R.A. Lafia-Araga, R. Yahaya and N.A. Rahman  
Department of Chemistry, University of Malaya, Malaysia
- MSD – 25 **COMPUTATION OF INTERFACIAL SHEAR STRENGTH AND OTHER TENSILE RELATED PROPERTIES OF INJECTION MOULDED CARBON FIBRE/POLYAMIDE 6,6**  
Rosiyah Yahya, Aziz Hassan, Zulkifli Abu Hasan  
Department of Chemistry, University of Malaya, 50603 Kuala Lumpur, MALAYSIA.
- MSD – 26 **IMPACT PROPERTIES OF INJECTION MOULDED ARAMID/CARBON HYBRID FIBRE REINFORCED POLYPROPYLENE COMPOSITES**  
MIM Rafiq and Aziz Hassan  
Department of Chemistry, University of Malaya, MALAYSIA
- MSD – 27 **HARDNESS OF CONTINUOUS HARD ANODIZING OF ALUMINIUM 6061 AFFECTED BY VOLTAGE AND TIME PROCESS USING TITANIUM CATHODE**  
Endi Sutikno, Ellen Desta Purwanendra, Putu Hadi Setyarini  
Mechanical Engineering Department, Brawijaya University, INDONESIA
- MSD – 28 **SURFACE CHARACTERISTICS OF ALUMINIUM HARD ANODIZING USING TITANIUM CATHODE**  
Putu Hadi Setyarini, Riviero Givenchy, Endi Sutikno  
Mechanical Engineering Department, Brawijaya University, INDONESIA

## PSE – PROCESS AND SYSTEM ENGINEERING

- [PSE – 01](#) **THE PROCESS TRANSPORT OF DRYING CORN WITH MIXED-ADSORPTION DRYING**  
Mohamad Djaeni, Luqman Buchori  
Chemical Engineering Department, Faculty of Engineering, Diponegoro University, INDONESIA
- [PSE – 02](#) **SIMULATION OF DUPLEX HEAT TREATMENT NB3SN COMPOUNDS IN CU-NB-SN SUPERCONDUCTING MULTIFILAMENTARY WIRE**  
Andini Nur Vania Swari, Arimaz Hangga, Doty Dewi Risanti  
Department of Engineering Physics, Faculty of Industrial Technology, ITS, INDONESIA

- PSE – 03** **INVENTORY ANALYSIS OF RADIOLOGICAL FOR GRAPHITE THERMAL COLUMN FROM TRIGA MARK II REACTOR, BANDUNG**  
Mulyono Daryoko  
*Radioactive Waste Technology Center, BATAN, INDONESIA*
- PSE – 04** **DYNAMIC SIMULATION AND COMPOSITION CONTROL IN A 10 L MIXING TANK**  
Yulius Deddy Hermawan  
*Chemical Engineering Department, Faculty of Industrial Technology, UPN “Veteran” Yogyakarta, INDONESIA*
- PSE – 05** **THE EFFECT OF LOADING ON THE CONTACT STRESS OF UHMWPE MATERIAL FOR ARTIFICIAL HIP JOINT BEARING**  
Eko Saputra<sup>a</sup>, Rifky Ismail<sup>a</sup>, Jamari<sup>a</sup> and Iwan Budiwan Anwar<sup>b</sup>  
<sup>a</sup> *Laboratory for Engineering Design and Tribology, Department of Mechanical Engineering, University of Diponegoro, INDONESIA*  
<sup>b</sup> *Orthopaedic Hospital dr. Soeharso, Solo, INDONESIA*
- PSE – 06** **SIMULATION OF COUNTER BLOW PROCESS OF PBL QUARTZ BOTTLE FABRICATION**  
Arimaz Hangga, Lizda Johar Mawarani, Doty Dewi Risanti  
*Department of Engineering Physics, Faculty of Industrial Technology, Institut Teknologi Sepuluh Nopember, INDONESIA*
- PSE – 07** **CADMIUM METALS PARTICLES-COVERED POLYSTYRENE NANOSPHERES THIN FILM MATERIAL:FABRICATION, ANALYSIS AND MODEL**  
Pratama Jujur Wibawa<sup>a,b,c</sup>, Hashim Saim<sup>d</sup>, Moh<sup>d</sup>. Arif Agam<sup>d</sup> and Hadi Nur<sup>e</sup>  
<sup>a</sup> *Microelectronic and Nanotechnology-Shamsuddin Research Center (MiNT-SRC), Universiti Tun Hussein Onn Malaysia, MALAYSIA*  
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<sup>e</sup> *Ibnu Sina Institute for Fundamental Science Studies, Universiti Teknologi Malaysia, MALAYSIA*
- PSE - 08** **DISTRIBUTION TEMPERATURE OF ANALYSIS ON CH4-CO2 GAS MIXED IN DOUBLE PIPE HEAT EXCHANGER BY CONTROLLED FEEZE OUT AREA METHODE**  
Fatma Y. Hasyim, Novi E. Mayangsari, and Sumarno  
Material of Laboratory  
*Chemical Engineering Department, Faculty of Industry Technology, Sepuluh Nopember Institut of Technology Surabaya, INDONESIA*
- PSE – 09** **SIMULATION ON THE ACTIVITY OF BIOMASS IN ACTIVATED SLUDGE IN THE PERFORMANCE OF NON IDEAL FLOW MEMBRANE BIOREACTOR SUBMERGED**  
Aisyah Endah Palupi  
*Mechanical Engineering Department, Engineering Faculty, State University of Surabaya 60231 – INDONESIA*
- PSE – 11** **EFFICACY AND HEAT TRANSFER EFFECTS OF ARTOCARPUS ALTILIS MALE INFLORESCENSE AS MOSQUITO VAPORIZING MATS**  
Muhammad Khamim Asy'Ari<sup>a</sup>, Nadhifa Maulida<sup>a</sup>, Wilujeng Fitri Alfiah<sup>a</sup>, Aisyiah Nur Isaneni<sup>a</sup> Titin Yulia Riska<sup>a</sup>, and Doty Risanti<sup>b</sup>  
<sup>a</sup> *Metrology and Instrumentation Diploma III Dept. of Engineering Physics Faculty of Industrial Engineering Institut Teknologi Sepuluh Nopember Surabaya, INDONESIA*  
<sup>b</sup> *Dept. of Engineering Physics Faculty of Industrial Engineering Institut Teknologi Sepuluh Nopember Surabaya, INDONESIA*

- PSE – 12     **SELECTION OF NATURAL DYE PHOTOSENSITIZER FOR QUASI-SOLID STATE DYE-SENSITIZED MESOPOROUS TiO<sub>2</sub> SOLAR CELL (DSC) FABRICATION**  
 Ruri Agung Wahyuono, Doty Dewi Risanti  
*Department of Engineering Physics, Institut Teknologi Sepuluh Nopember Surabaya, INDONESIA*
- PSE – 13     **AN INVESTIGATION OF CHAR FORMATION AND SHRINKING VOLUME BY VISUALIZATION TECHNIQUE INDUCED BY PYROLYSIS**  
 Widya Wijayanti  
*Mechanical Engineering Department, Brawijaya University, INDONESIA*
- 

## SPE – SEPARATION PROCESS ENGINEERING

- [SPE – 01](#)     **UTILIZATION OF COAL FLY ASH AS CO GAS ADSORBENT**  
 Ayu Lasryza, Dyah Sawitri  
*Department of Engineering Physics, Faculty of Industrial Technology, ITS, INDONESIA*
- [SPE – 02](#)     **PHENOL REMOVAL FROM AQUEOUS SOLUTIONS BY ELECTROCOAGULATION TECHNOLOGY USING IRON ELECTRODES: EFFECT OF SOME VARIABLES**  
 Mohammad Ali Zazouli<sup>a</sup>, Mahmoud Taghavi<sup>b</sup>  
<sup>a</sup> *Department of Environmental Health Engineering, Faculty of Health and Health Sciences Research Center, IRAN*  
<sup>b</sup> *Department of Environmental Health Engineering, Faculty of Health and Health Sciences Research Center, IRAN*
- [SPE – 03](#)     **THE OFF GAS TREATMENT IN THE PROCESS OF VITRIFICATION AND INCINERATION OF NUCLEAR WASTE**  
 Herlan Martono, Aisyah  
*Radioactive Waste Technology Centre, BATAN, INDONESIA*
- [SPE – 04](#)     **ESSENTIAL OIL EXTRACTION OF FENNEL SEED (FOENICULUM VULGARE) USING STEAM DISTILLATION**  
 Astrilia Damayanti and Eko Setyawan  
*Chemical Engineering Department, Faculty of Engineering, Semarang State University, INDONESIA*
- [SPE – 05](#)     **PREPARATION AND CHARACTERIZATION OF NANOFILTRATION MEMBRANE FOR WATER TREATMENT**  
 Tutuk Djoko Kusworo, Eka Cahya Muliawati and Ardian Dwi Yudhistira  
*Department of Chemical Engineering, Diponegoro University, INDONESIA*
- [SPE – 06](#)     **THE APPLICATION OF NITROGEN LASER ON EXTRACTION OF URANIUM IN THE LONG LIFE OF HIGH LEVEL RADIOACTIVE LIQUID WASTE USING TBP-KEROSENE SOLVENT**  
 Gunandjar  
*Radioactive Waste Technology Center, National Nuclear Energy Agency of Indonesia (BATAN), INDONESIA*
- [SPE – 07](#)     **EFFECT OF PH AND STIRRING SPEED ON THE COLLAGENOUS PROTEIN EXTRACTION FROM CHICKEN BONE WASTE IN A WELL AGITATED EXTRACTION SYSTEM**  
 Andri Cahyo Kumoro, Beatrice L. M. Tanjung, Fadilla H. Utami, Diah Susetyo Retnowati and Catarina Sri Budiayati  
*Department of Chemical Engineering, Diponegoro University, INDONESIA*
- [SPE – 08](#)     **DRYING OF CURCUMA (CURCUMA XANTHORRHIZA ROXB) USING DOUBLE PLATE COLLECTOR SOLAR DRYER**  
 Tjukup Marnoto, Mahreni, Wasir Nuri, Bayu Ardinanto, Ratna E. Puspitasari  
*Chemical Engineering Department, Faculty of Industrial Technology, UPN "Veteran" Yogyakarta University, INDONESIA*

- SPE – 09**      **SOLUBILITY EXAMINATION OF PALM KERNEL OIL IN SUPERCRITICAL CO<sub>2</sub> AND ITS CORRELATION WITH SOLVENT DENSITY BASED MODEL**  
 Wahyu Bahari Setianto<sup>a</sup>, Priyo Atmaji<sup>b</sup> and Didi Dwi Anggoro<sup>b</sup>  
<sup>a</sup> Center for Agroindustrial Technology, Agency for the Assessment and Application of Technology, INDONESIA  
<sup>b</sup> Department of Chemical Engineering, Diponegoro University, INDONESIA
- SPE – 10**      **Oil Extraction Process From Solid Waste Rubber Seed By Soxhletation and Extraction Solvent by Stirring Methods**  
 Achmad Wildan, Devina Ingrid A, Indah Hartati  
 Sekolah Tinggi Ilmu Farmasi “Yayasan Pharmasi” Semarang  
 Jl. Sarwo Edhie Wibowo Km. 1 Plamongsari, Pucanggading Semarang INDONESIA
- SPE – 11**      **FOULING AND REJECTION BEHAVIOUR OF ULTRAFILTRATION FOR OIL IN WATER EMULSION SEPARATION**  
 Nita Aryanti<sup>a</sup>, Agus Hadiyanto<sup>a</sup>, Wiharyanto Oktiawan<sup>b</sup>  
<sup>a</sup> Chemical Engineering Department, Diponegoro University, INDONESIA  
<sup>b</sup> Environmental Engineering Department, Diponegoro University, INDONESIA
- SPE – 12**      **THE EFFECT OF CHEMICAL ADDITIVES ON MOBILITY OF HEAVY METALS (PB, CD AND ZN) IN SOIL**  
 Abdoliman Amouei<sup>a</sup>, Amirhossein Mahvi<sup>b</sup>, Masoumeh Tahmasbizadeh<sup>b</sup>, Mohammad Ali Zazouli<sup>c</sup>  
<sup>a</sup> Department of Environmental Health Engineering, Babol University of Medical Sciences, IRAN  
<sup>b</sup> Department of Environmental Health Engineering, Tehran University of Medical Sciences, IRAN  
<sup>c</sup> Department of Environmental Health Engineering, Faculty of Health and Health Sciences Research Center, Mazandaran University of Medical Sciences, IRAN
- SPE – 13**      **CHARACTERISTICS OF HOSPITAL WASTEWATER IN BABOL UNIVERSITY OF MEDICAL SCIENCES AND EFFECTS ON THE ENVIRONMENT**  
 Abdoliman Amouei<sup>a</sup>, Hosseinali Asgharnia<sup>a</sup>, Hourieh Fallah<sup>a</sup>, Aliakbar Mohammadi<sup>a</sup>, Mohammad Ali Zazouli<sup>b</sup>  
<sup>a</sup> Department of Environmental Health Engineering, Babol University of Medical Sciences, IRAN.  
<sup>b</sup> Department of Environmental Health Engineering, Faculty of Health and Health Sciences Research Center, Mazandaran University of Medical Sciences, Sari, IRAN
- SPE – 14**      **QUANTITY AND QUALITY OF SOLIDWASTES IN THE HOSPITALS OF BABOL UNIVERSITY OF MEDICAL SCIENCES, NORTH OF IRAN**  
 Abdoliman Amouei<sup>a</sup>, Masoumeh Tahmasbizadeh<sup>b</sup>, Hosseinali Asgharnia<sup>a</sup>, Hourieh Fallah<sup>a</sup>, Aliakbar Mohammadi<sup>a</sup>, Mohammad Ali Zazouli<sup>c</sup>  
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<sup>b</sup> Department of Environmental Health Engineering, Tehran University of Medical Sciences, IRAN  
<sup>c</sup> Department of Environmental Health Engineering, Faculty of Health and Health Sciences Research Center, Mazandaran University of Medical Sciences, IRAN
- SPE – 15**      **EVALUATION OF PHENOL REMOVAL FROM AQUEOUS SOLUTIONS BY AZZOLA**  
 Davaud Balarak<sup>a</sup>, Mohammad Ali Zazouli<sup>b</sup>  
<sup>a</sup> University Mazandaran – Sari, IRAN  
<sup>b</sup> Department of Environmental Health Engineering, Faculty of Health and Health Sciences Research Center, Mazandaran University of Medical Sciences, IRAN
- SPE – 16**      **SILICA EXTRACTION FROM BAGGASE FLY ASH WITH ALKALI FUSION METHOD**  
 Galuh Pinayungan, Arif Hidayat, Chandra Wahyu Purnomo, Arief Budiman  
 Process System Engineering Research Group, Chemical Engineering Department, Gadjah Mada University, INDONESIA

Cover	1
Scientific and Editorial Board Members	2
Editor	3
Organizing Committee	4
Preface	6
Contents	7
Table of Abstracts	8
Programme Schedule	17
Abstract	
Invited Speakers	21
Oral Presentation	
Bioprocess and Renewable Energy (BRE)	27
Material and Science Development (MSD)	52
Process System Engineering (PSE)	80
Separation and Process Engineering (PSE)	92
Acknowledgement	



# Optimization Process of Biodiesel Production From Nyamplung Seed (*Calophyllum inophyllum* L) Using In Situ Process and Ultrasonic Assisted

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## Abstract :

Nyamplung seed (*Calophyllum inophyllum*) contain high oil between 40-73%, so it potential as a raw material in biodiesel production. It The objective of this research to optimization of biodiesel production by in situ process and assisted ultrasonic. Optimization process use central composite design methods. Variables were investigated temperature, catalyst concentration (methanol basis), and the weight ratio of methanol by weight nyamplung. The ultrasonic wave was produced by ultrasonic cleaner unit with frequency 40 KHz, degassing time 10 minutes and reaction time 60 minutes. The results of research obtained Optimum conversion of biodiesel production from nyamplung seed with assisted ultrasonic was 64.429%. The optimum condition are 1:5.9116, 2.556% and 46.2°C for ratio weight of nyamplung seed to

$$Y = 69.609 - 0.7586X_1 + 1.9051X_2 + 3.2476X_3 - 2.08X_1X_2 - 3.665X_1X_3 - 2.865X_2X_3 - 10.6085X_1^2 - 6.224X_2^2 - 6.747X_3^2$$
methanol, catalyst concentration and temperature respectively. Mathematic modeling for describe in this process like expressed;

**Keywords:** biodiesel, conversion, nyamplung (*Calophyllum Inophyllum*), ultrasonic assisted

## 1. Introduction

Fossil fuel consumption continues to raise nationwide average 7% per year, estimated in 2020 the consumption of fossil fuels reached 34 million kilo liters. Consumption of fossil fuels percentage is the largest and constantly increasing. In 2008 there was a surge in crude oil around \$ 1000/barrel, this is causing a crisis in the world and in Indonesia. World oil prices in 2011 USD 107.46/barrel and in 2012 USD 110.27/barrel [1]. The increase in oil prices could encourage the energy crisis is more severe, so that alternative energy is needed to overcome.

Indonesia government efforts to solve energy crisis is with the establishment of policies. Indonesia government policies set out in Presidential Decree no. 5 of 2006 regarding the national energy policy, Presidential Instruction No. 1 of 2006 regarding the procurement and use of biofuels as alternative energy, as well as the Presidential Decree no.10 of 2006 regarding the formation of a national policy for the development of biofuels [2].

Indonesia has great potential to produce alternative energy instead of fossil fuels derived from renewable natural resources in the form of plants. Vegetable oil (vegetable) and animal fats as an alternative energy source can be processed into biodiesel. Biodiesel is a methyl ester compound result from the transesterification reaction of triglycerides derived from vegetable oils or animal fats [3-6]. Biodiesel has several advantages if compared with conventional fuel. The advantages are environmentally friendly, good lubricating power, fewer emissions and a relatively clean burning character. In addition to these advantages, the use of biodiesel also provides benefits to the vehicle engine maintenance [7,8]. Processing of biodiesel from renewable raw materials have been widely applied in various countries including countries of the United States uses soybean oil, coconut oil (coconut oil) in the Philippines and Malaysia using CPO (Crude Palm Oil). Indonesia uses Crude Palm Oil, nyamplung oil and castor oil (*Jatropha*) for raw material in biodiesel production.

*Calophyllum inophyllum* L (nyamplung) is a medium-sized to large evergreen tree that averages 8–20 m (25–65 ft) in height with a broad spreading crown of irregular branches (like Fig 1). The tree supports a dense canopy of glossy, elliptical leaves, fragrant white flowers, and large round nuts. It grows along coastal areas and adjacent lowland forests, although it occasionally occurs inland at higher elevations. It is native to east Africa, India, Southeast Asia, Australia, and the South Pacific. It has been widely planted throughout the tropics and is naturalized in the main Hawaiian islands. One large brown seed 2–4 cm (0.8–1.6 in) in diameter is found in each

fruit. Seeds are prepared by cleaning off the skin and husk from the shell of the seed; there are 100–200 seeds/kg (45–90 seeds/lb), with shells intact but husks removed [9,10]. Nyamplung seed figure like presented in figure 2.



**Figure 1.** Nyamplung tree and seed



**Figure 2.** Ripe fruit (left), cracked shell showing seed kernel inside (middle), and dry seed (right).

Biodiesel from nyamplung seed (*Calophyllum inophyllum* L) is one of the alternative fuels from vegetable oils other than castor oil, palm oil, sunflower seeds, and used frying oil (UFO). Nyamplung plants (*Calophyllum inophyllum*) is one of the alternative feedstock biodiesel that has considerable potential. Nyamplung can produce oil per year at 20 tons / ha. Nyamplung seed contain high oil between 40-73%. One liter of nyamplung oil can be produced from 2.5 kg of nyamplung seed, while the castor requires 4 kg to produce one liter of oil [10,11].

The main reaction to produce biodiesel is esterification and transesterification reactions [3-8]. In biodiesel production can be done with the conventional method and the method of ultrasonic waves. The process of making the conventional method relatively slow (almost 4-5 hrs), need more catalyst and alcohol and furthermore the reaction is not fully completed [5]. Moreover, the high formation of by-product gives low yield of biodiesel and during the conversion process not all fatty acid chains are turned into biodiesel which consequently reduces the quality and the yield. The current conventional process only can achieve maximum yield of 85% during esterification reaction [4-6]. Therefore, an alternative process is really required to reduce the processing time, to increase the yield, to lower amount un-reacted methanol and also catalyst, and to increase the mass transfer.

Ultrasound (~20 kHz to 10 MHz ) is cyclic sound pressure with a frequency greater than the upper limit of human hearing. The important use of ultrasound is to create bubble cavitations which can be used as replacement of mechanical agitation by stirrer, and therefore it is required for enhancing the esterification reaction. This paper describes the potential use of ultrasound for assisting biodiesel production using nyamplung seed In biodiesel production by assisted ultrasonic wave takes up to 1 hour [12-13].

Ultrasonic technology has advantages like minimum energy consumption and can be reduced time operation. So, these technologies promising because it offers the potential for shorter reaction cycles leading to the formation of chemical plants are smaller and cheaper. Excellence performance using ultrasonic solvent extraction method compared to conventional methods has also been reported by several researchers [13-15].

## 2. Material and Methods

### 2.1. Apparatus and Materials

Raw materials using in this experiment are nyamplung seed collected from Banyumas, Central Java. Nyamplung seed crushed into powder. Methanol used has industrial grade and  $H_2SO_4$  as catalyst has analytical grade from MERCK. Analytical chemical was used like as; ethanol 95% (technical grade), KOH (analytical grade), HCl (analytical grade), and Dietil Eter (analytical), PP indicator. Chemicals have analytical grade obtained from MERCK. Apparatus using in this experiment are ultrasonic cleaner according to BRANSON, aluminum foil, 250 ml Erlenmeyer PYREX. The experimental set up shown in Figure 3. Ultrasonic cleaner has frequency 40 kHz.



Figure 3. Schematic diagram of the ultrasonic cleaner

### 2.2. Experimental Procedure

25 grams of nyamplung seed powder mixed with methanol and sulphuric acid as catalyst in erlenmeyer. Then, the Erlenmeyer is inserted into the round pedestal ultrasonic cleaner. Operate the ultrasonic cleaner for 60 minutes at a predetermined variable. Erlenmeyer took from the ultrasonic cleaner and then cooled to room temperature. The mixture is distilled to separate the biodiesel with methanol. Variables used in this experiments like presented in Table 1. Fixed variable was used weight nyamplung seed powder 25 grams, size 200 mesh powder nyamplung, frequency of 40 kHz ultrasonic waves, reaction time of 60 minutes, set Degas 10 minutes.

### 2.3. Analysis Method

Product was analysis of volume of product, number of acid, viscosity, density and gas chromatography. Volume and density of product was used for yield calculation. Yield of biodiesel was calculated with equation 1. Yield of biodiesel will be processed by the Central Composite Design (CCD) method to evaluate the response and the observation of the influential variables. The analysis process used Statistica 6 software about mathematical model, statistic analysis, analysis of variance and profile of yield versus variable processes. The experiment then compared with conventional processes.

$$\text{Yield (\%)} = \frac{\text{weight of biodiesel}}{\text{weight of nyamplung seed}} \times 100 \quad [1]$$

## 3. Result and Discussion.

### 3.1. Experiments Data

The results of data processing are presented in Table 1. Central Composite Design used 2 blocking, 3 variable processes. The experiment 1-9 classified in 1 blocking and 10-16 classified in 2 blocking.  $X_1$  is coding of ration weight of nyamplung seed to methanol volume.  $X_2$  is coding of catalyst concentration (methanol based).  $X_3$  is coding of temperature operation. The value that used for experiment can be calculated with equation 2-4. The real condition of variable can be calculated with equation 2-4.

**Table 1.** The result and experiments design of CCD methods

Run	Block	X <sub>1</sub>	X <sub>2</sub>	X <sub>3</sub>	ρ	Y (%)
1.	1	-1	-1	-1	0.880	29.16
2.	1	-1	-1	+1	0.881	55.28
3.	1	-1	+1	-1	0.902	48.56
4.	1	-1	+1	+1	0.883	42.48
5.	1	+1	-1	-1	0.891	46.92
6.	1	+1	-1	+1	0.882	37.64
7.	1	+1	+1	-1	0.883	37.26
8.	1	+1	+1	+1	0.884	37.26
9.	1	0	0	0	0.892	51.88
10.	2	-1.68	0	0	0.885	38.92
11.	2	+1.68	0	0	0.887	42.12
12.	2	0	-1.68	0	0.889	45.44
13.	2	0	+1.68	0	0.905	62.76
14.	2	0	0	-1.68	0.889	42.44
15.	2	0	0	+1.68	0.889	62.52
16.	2	0	0	0	0.888	86.96

Where X<sub>1</sub> : (-1) 1:5 (0) 1:6 and (+1) 1:7  
X<sub>2</sub> : (-1) 2% (0) 2.5% and (+1) 3%  
X<sub>3</sub> : (-1) 40 ° C, (0) 45 ° C, and (+1) 50 ° C.  
(-1) : below value (bv)  
(0) : central value (cv)  
(+) : upper value (uv)

$$X_1 = \frac{[R-6]}{1} \quad [2]$$

$$X_2 = \frac{[K-2.5]}{0.5} \quad [3]$$

$$X_3 = \frac{[T-45]}{5} \quad [4]$$

### 3.2. Statistics Analysis

The results of statistic analysis include mathematical model, t test, analysis of variance, pareto analysis and validation of mathematical model. Polynomial equations to mathematical model the CCD as follows:

$$Y_u = \beta_0 + \sum \beta_i X_{ui} + \sum \beta_{ii} X_{ui}^2 + \sum \sum \beta_{ij} X_{ui} X_{uj} + \varepsilon \dots \dots \dots (5)$$

Where:

Y<sub>u</sub> = predicted response to u, u: 1, 2, 3, ....., n

β<sub>0</sub> : average of yield

β<sub>i</sub>: linear coefficient, β<sub>ii</sub>: squared term coefficient, β<sub>ij</sub>: interactions variable coefficient

xi: non dimensional number of independent variables

In this experiment the number of independent variables was 3, so the polynomial equation becomes

$$Y_u = \beta_0 + \beta_1 X_1 + \beta_2 X_2 + \beta_3 X_3 + \beta_{12} X_1 X_2 + \beta_{13} X_1 X_3 + \beta_{23} X_2 X_3 + \beta_{11} X_1^2 + \beta_{22} X_2^2 + \beta_{33} X_3^2 \quad [6]$$

The results of analysis regression for equation 6 like presented in equation 7. Data that used for this analysis presented in table 1. In the mathematical model shows that the coefficient of linear variable-value range, most of the variables X<sub>3</sub> and X<sub>1</sub> and X<sub>2</sub> variables followed. Variables X<sub>2</sub> and X<sub>3</sub> are positive which means the larger value of this variable, and then the yield of biodiesel obtained the greater. The first variable is the inverse of the variables X<sub>2</sub> and X<sub>3</sub>. Increased temperatures will lead to increasing the solubility also increases the reaction rate. Increasing of catalyst concentration can enhance the catalytic activity of direct transesterification reaction, thereby increasing the amount of methyl ester.

$$Y = 69.609 - 0.7586 X_1 + 1.9051 X_2 + 3.2476 X_3 - 2.08 X_1 X_2 - 3.665 X_1 X_3 - 2.865 X_2 X_3 - 10.6085 X_1^2 - 6.224 X_2^2 - 6.747 X_3^2 \quad [7]$$

Coefficient values in equation 7, further evaluated the t test and its variations. For the t test using an  $\alpha = 0.05$ . The results of such analysis are presented in Tables 2 and 3. In Table 2 show that the coefficients of variables  $X_2$  and  $X_3$  have a value of t (5) is greater than the value of p, but the value of t (5) is still in the range t test.

**Table 2.** Results of regression coefficient and t test

Factor parameter	Regression coef	Standar error	t(5)	p	-95% Cnf. Limnt	+95% Cnf. Limnt
Mean	69.6093	8.557627	8.13419	0.000456	47.6113	91.60741
Blocking	5.7113	3.049887	1.87263	0.120007	-2.1287	13.55130
$X_1$	-0.7586	3.212522	-0.23613	0.822701	-9.0166	7.49949
$X_1^2$	-10.6085	3.757510	-2.82327	0.036966	-20.2674	-0.94947
$X_2$	1.9051	3.212522	0.59302	0.578971	-6.3530	10.16315
$X_2^2$	-6.2244	3.757510	-1.65653	0.158516	-15.8834	3.43457
$X_3$	3.2476	3.212522	1.01093	0.358442	-5.0104	11.50568
$X_3^2$	-6.7474	3.757510	-1.79571	0.132487	-16.4064	2.91158
$X_1 X_2$	-2.0800	4.279292	-0.48606	0.647476	-13.0803	8.92027
$X_1 X_3$	-3.6650	4.279292	-0.85645	0.430872	-14.6653	7.33527
$X_2 X_3$	-2.8650	4.279292	-0.66950	0.532843	-13.8653	8.13527

**Table 3.** Result of analysis of variance

Factor parameter	SS	Degree of Freedom	MS	F	p
Blocking	513.734	1	513.734	3.506746	0.120007
$X_1$	8.168	1	8.168	0.055756	0.822701
$X_1^2$	1167.718	1	1167.718	7.970840	0.036966
$X_2$	51.520	1	51.520	0.351674	0.578971
$X_2^2$	402.004	1	402.004	2.744082	0.158516
$X_3$	149.718	1	149.718	1.021978	0.358442
$X_3^2$	472.397	1	472.397	3.224578	0.132487
$X_1 X_2$	34.611	1	34.611	0.236256	0.647476
$X_1 X_3$	107.458	1	107.458	0.733507	0.430872
$X_2 X_3$	65.666	1	65.666	0.448235	0.532843
Error	732.494	5	146.499		
Total SS	2894.021	15			

Pareto diagram is histogram of the data is sorted based on categories of greatest to smallest. It is based on the principle that there are many factors that affect anything but just a few important factors are taken into account the factors that cause the most significant impact. Thus, pareto diagrams can assist in focusing efforts on the most important thing alone [17], [18]. Pareto diagram of data processing results in this study are presented in Figure 4. Pareto analysis results showed that a linier variable of  $X_1$  has smaller value. So this variable can be neglected in optimization process. Pareto chart show variable of ratio nyamplung to methanol (Q) has a histogram that excess the line  $p = 0.05$ . The others variable have histogram don't cross the line  $p = 0.05$ .

Mathematical model in 7 equations was validated with experiments data. The results shown in Figure 5. Mathematical models that is less valid because the experimental data coincide with the results of the calculation very little.

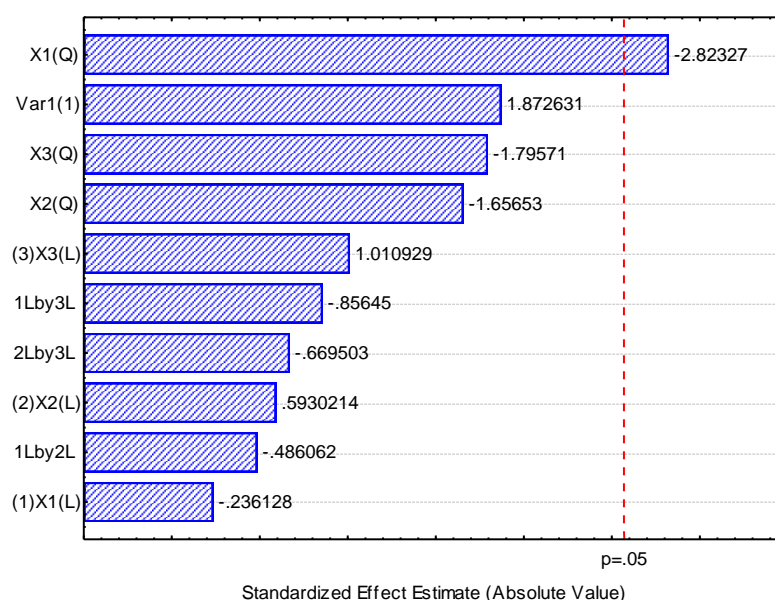


Figure 4. Pareto chart

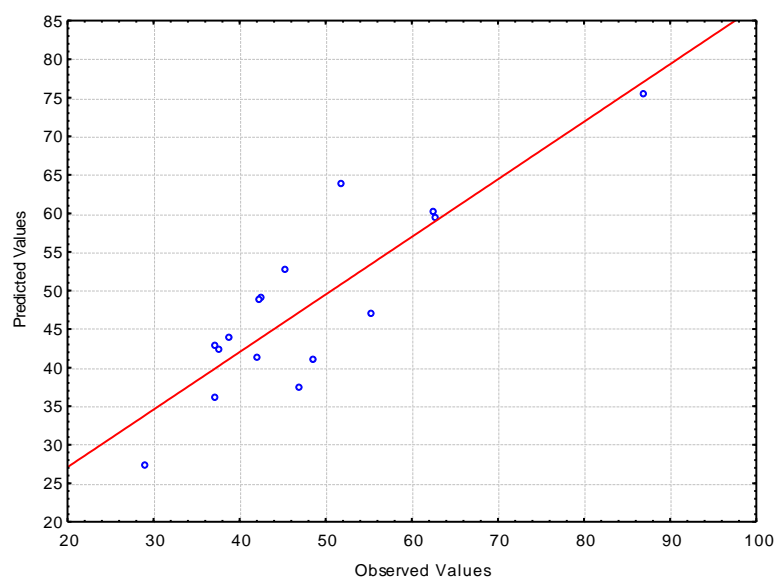


Figure 5. Graph of validation model mathematics

### 3.3. Optimization Results

Optimum operating conditions sought by looking at optimization 3-dimensional graph and surface contours graph. Optimization of three-dimensional graph consists of axis x, y, and z, where x and y axis is the variable being tested while the z-axis shows the value of the conversion can be achieved from the interaction of two variables tested so from optimization of three-dimensional graphics can be seen the value of the conversion which can be achieved from the interaction of two variables that are tested and as well as optimal conditions. Surface contour graph consists of axis x and y, where x and y axes are the variables tested. In surface contours figuring in color areas, so it can be seen from this graph the points of interaction of two variables is clear, where most interactions are optimal in the red region of the oldest.



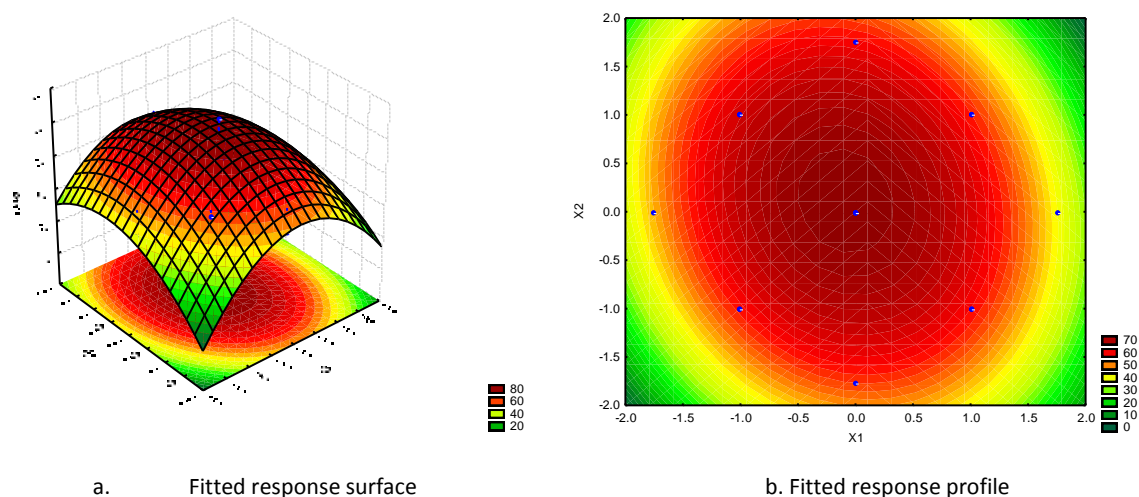


Figure 6. Yield versus  $X_1$  and  $X_2$

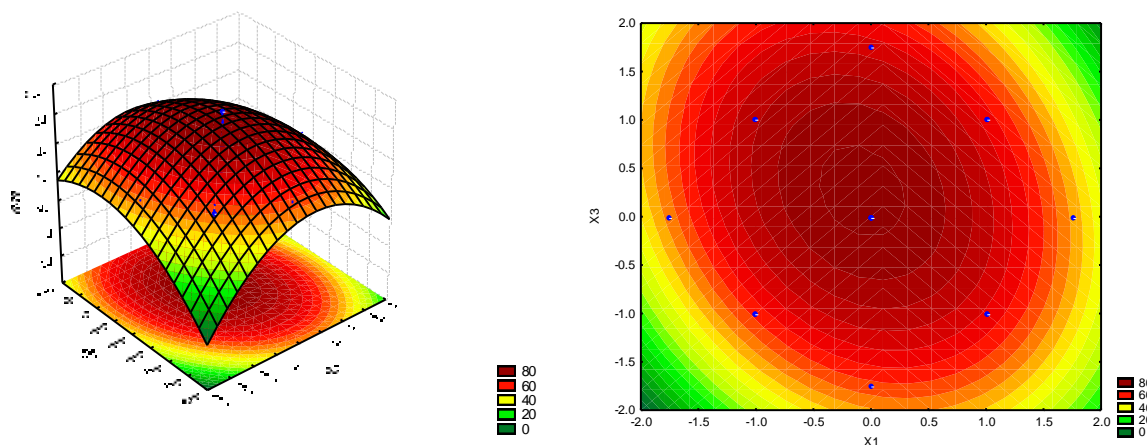


Figure 7. Yield versus  $X_1$  and  $X_2$

The results of the response fitted surface graphs are parabolic and contour plot is shaped oval. This suggests that the type of optimization process is already maximized. The critical value for each variable is shown in the following table:

Table 2. Critical value each variables

No.	Variable	Observed Minimum	Critical Values	Observed Maximum
1.	Ratio of Nyamplung to Methanol	-1.76000	-0.0884	1.760000
2.	Catalyst concentration	-1.76000	0.1124	1.760000
3.	Temperature	-1.76000	0.2408	1.760000

In table 2, the critical value of dimensionless numbers for each variable. Critical dimensionless value obtained for  $X_1$  (weight ratio nyamplung to methanol) is -0.0884,  $X_2$  (catalyst concentration) 0.1124 and  $X_3$  (temperature operation) is 0.2408. These values were input in 7 equations and obtained conversion of biodiesel is 64.429%. These values were intake in 2-4 equation ad obtained optimum condition are 1:5.9116, 2.556% and 46.2oC for ratio weight of nyamplung seed to methanol, catalyst concentration and temperature respectively.

### 3.4. Biodiesel Production with Assisted Ultrasonic versus Conventional Process

**Table 4.** Characteristics biodiesel with ultrasonic and conventional

No.	Characteristics	Conventional	Ultrasonic wave
1.	Density	0.932	0.888
2.	Kinematic viscosity (Cst)	4.88	4.5
3.	Conversion	54.09	86.96

Table 4 shows the processed biodiesel that processed with ultrasonic waves have better characteristics than conventionally processed biodiesel. With reaction time and the same reaction process variables biodiesel conversion that resulting from ultrasonic wave is greater than the conventional processes. The rate of transesterification reaction using ultrasonic waves higher than the use of a mechanical stirrer [14,15]. This is because the effects of cavitations and oscillations of the use of ultrasonic sound waves in the transesterification reaction. Oil molecules or FFA that reacted with methanol has a very fast movement and bounced hard. The movement of oil and methanol molecules is random, so that the stirring becomes more homogeneous. The micro bubbles of storing large energy in the form of surface tension, where the magnitude of surface tension is inversely proportional to the radius of the bubble. Small diameter of the bubbles that will save energy in the form of a large surface tension [12,13].

### 3.5. Biodiesel Characteristics

**Table 5.** Characteristics experiment biodiesel and SNI standard [16]

No.	Characteristics	Biodiesel from experiment	SNI standard
1.	Density (g/ML)	0,888	0,85-0,89
2.	Viscosity (cSt)	4,5	2,3-6,0
3.	Acid number (mg KOH/g)	0,44	Max 0,8

Table 5 shows that the characteristics of the biodiesel meets the characteristics of the experimental results already established by the SNI. Calophyllum inophyllum-biodiesel viscosity slightly higher but still meet the standards set by SNI. Oil viscosity is expressed by the number of time (seconds) used by a certain volume of oil to flow through small diameter holes with a particular, the lower the second number means that the lower the viscosity. Viscosity is too high can the burden of the pump and cause poor fogging [8].

The density of biodiesel from nyamplung oils meet the requirements of SNI biodiesel is 0.888 g / ml. Although the density of biodiesel is slightly higher, but still included in the SNI standard biodiesel making biodiesel produced viable. Differences related to the density of biodiesel fatty acid composition and degree of purity of the biodiesel [7].

## 4. Conclusion

Biodiesel derived from the seeds of raw materials Nyamplung (*Calophyllum inophyllum*) is in conformity with the SNI. This is indicated by the physical and chemical properties of biodiesel. Optimum conversion of biodiesel production from nyamplung seed with assisted ultrasonic was 64.429%. The optimum condition are 1:5.9116, 2.556% and 46.2oC for ratio weight of nyamplung seed to methanol, catalyst concentration and temperature respectively. Model equations for the biodiesel formation reaction is:

$$Y = 69.609 - 0.7586X_1 + 1.9051X_2 + 3.2476X_3 - 2.08X_1X_2 - 3.665X_1X_3 - 2.865X_2X_3 - 10.6085X_1^2 - 6.224X_2^2 - 6.747X_3^2$$

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*This is to certify*  
**Widayat**

as presenter

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